

[\[Return to Article\]](#)**Table 1.** Multidimensional NMR experiments

Experiment (acquisition of central peaks)*	Indirect [†] dimension(s)	t_{\max} , ms; complex points	Measurement time, h	Relative sensitivity (peak pairs/ central peaks)
Sequential backbone connectivities (3D spectra)				
$\underline{H^{\alpha/\beta}}\underline{C^{\alpha/\beta}}(\text{CO})\text{NHN}$	$\omega_1(^{13}\text{C}^{\alpha/\beta}/^1\text{H}^{\alpha/\beta})$	6.3; 95	9.2	(0.56/0.34)
(^{13}C)	$\omega_2(^{15}\text{N})$	21.5; 28		
$\underline{HACA}(\text{CO})\text{NHN}$	$\omega_1(^{13}\text{C}^{\alpha}/^1\text{H}^{\alpha})$	6.5; 54	5.4	(1.00[‡] /0.81)
(^{13}C)	$\omega_2(^{15}\text{N})$	21.5; 28		
Intraresidual backbone connectivities (3D spectra)				
$\underline{\text{HNNCAHA}}$	$\omega_1(^{13}\text{C}^{\alpha}/^1\text{H}^{\alpha})$	6.6; 51	5.0	(0.41/0.27)
(INEPT)	$\omega_2(^{15}\text{N})$	21.5; 28		
$\underline{H^{\alpha/\beta}}\underline{C^{\alpha/\beta}}\text{COHA}$	$\omega_1(^{13}\text{C}^{\alpha/\beta}/^1\text{H}^{\alpha/\beta})$	6.3; 95	10.0	(0.22/0.11)
(^{13}C)	$\omega_2(^{13}\text{C}=\text{O})$	17.8; 32		
$\underline{\text{HNNCACB}}$	$\omega_1(^{13}\text{C}^{\alpha/\beta})$	6.6; 56	8.0	(0.56)
	$\omega_2(^{15}\text{N})$	21.5; 28		
Intra- and sequential-backbone connectivities (3D spectrum)				
$\underline{\text{HNN}(\underline{\text{CO}},\underline{\text{CA}})}$	$\omega_1(^{13}\text{C}^{\alpha}/^{13}\text{C}=\text{O})$	8.0/16.0 [§] ; 54	5.5	(0.54/1.41)
(INEPT)	$\omega_2(^{15}\text{N})$	21.5; 28		
Assignment of aliphatic resonances (3D spectra)				
$\underline{\text{HCCH}}\text{-COSY}$	$\omega_1(^{13}\text{C}/^1\text{H})$	6.3; 95	6.2	(0.34/0.25)
(^{13}C)	$\omega_2(^{13}\text{C})$	6.4; 20		
$\underline{\text{HCCH}}\text{-TOCSY}^{\ddagger}$	$\omega_1(^{13}\text{C}/^1\text{H})$	6.3; 95	7.0	(0.19/n.d.)
(^{13}C)	$\omega_2(^{13}\text{C})$	6.4; 20		

Assignment of aromatic resonances (2D spectra)

<u>HBCB</u> (CGCD)HD	$\omega_1(^{13}\text{C}/^1\text{H})$	6.3; 95	5.3	(0.45/0.33)
(^{13}C)				
^1H -TOCSY- <u>HCH</u> -COSY [†]	$\omega_1(^{13}\text{C}/^1\text{H})$	15; 150	3.4	(0.76/-)

One millimolar solution of "Z-domain" of *Staphylococcal* protein A at T = 25°C. The radio-frequency (rf) carrier for ^1H -frequency labeling in the projected "HC"-dimensions in which the chemical shifts of the aliphatic moieties are measured was set to 0 ppm. In 2D ^1H -TOCSY-HCH-COSY, the ^1H rf carrier was set to the position of the water line throughout. t_{max} denotes the maximal evolution time.

The suite of experiments in this table can provide complete resonance assignments of proteins, excluding only the side chain NH_n moieties, the CH^r groups of histidiny, and the CH^{*3} , $\text{CH}^{*2,3}$, and CH^{*2} groups of tryptophanyl residues (which can be obtained as described in ref. 17). Notably, Z-domain does not contain tryptophans.

* Approach 1: Use of incomplete polarization transfer (rows labeled with "INEPT"); Approach 2: use of ^{13}C steady state magnetization (rows labeled with " ^{13}C ").

† Direct dimension: $t_{\text{max}} = 73$ ms/512 complex points.

‡ The average signal-to-noise (S/N) ratio of peaks observed in this subspectrum was 33.2.

§ The increment for $^{13}\text{C}^\alpha$ chemical shift evolution was scaled by a factor of 0.5 relative to the values used to sample $^{13}\text{C}=\text{O}$ evolution (5).

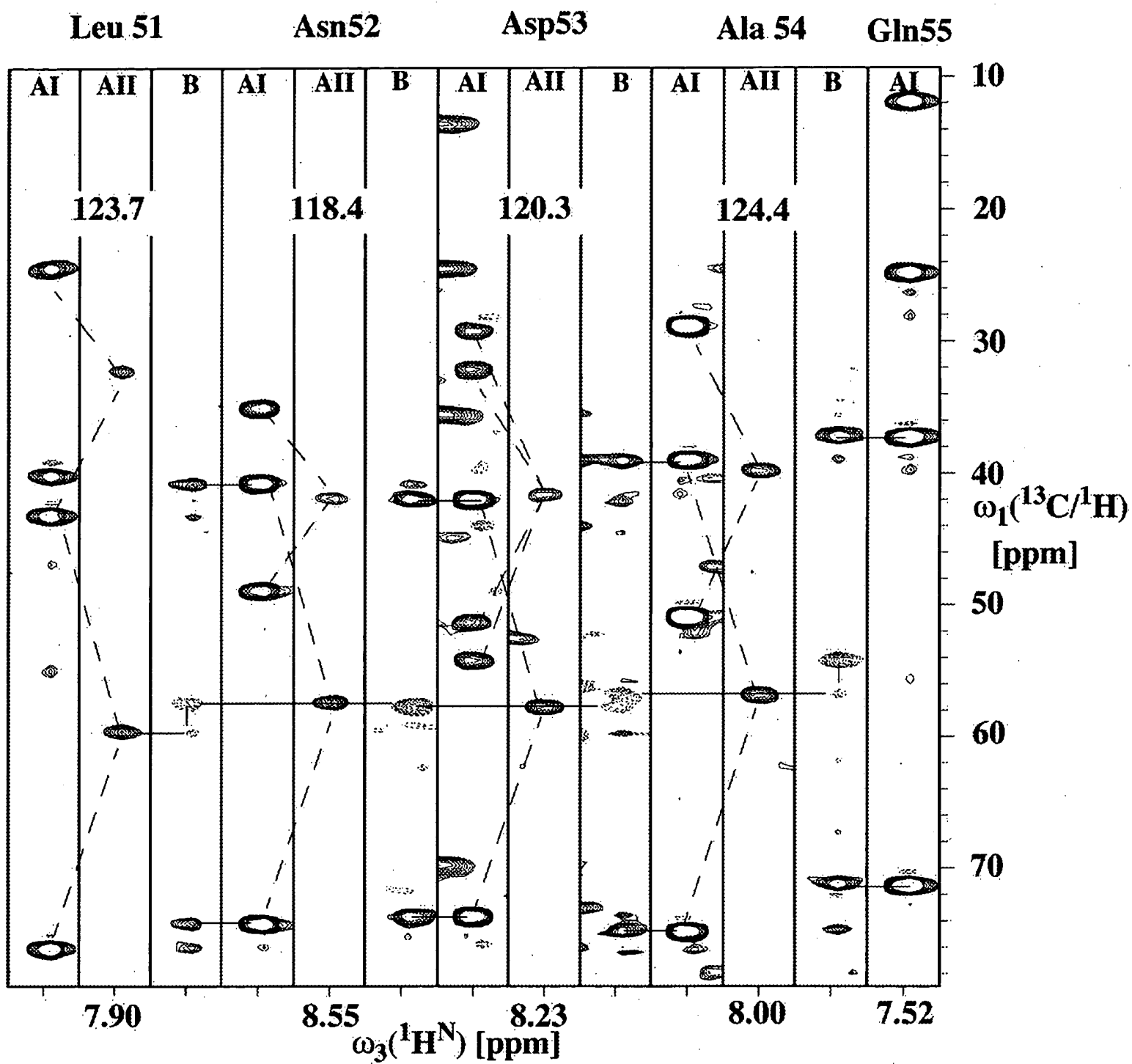
¶ The mixing times for the ^{13}C -TOCSY relay was set to 21 ms. The S/N ratios for the double-relay central peaks were too low to be accurately evaluated.

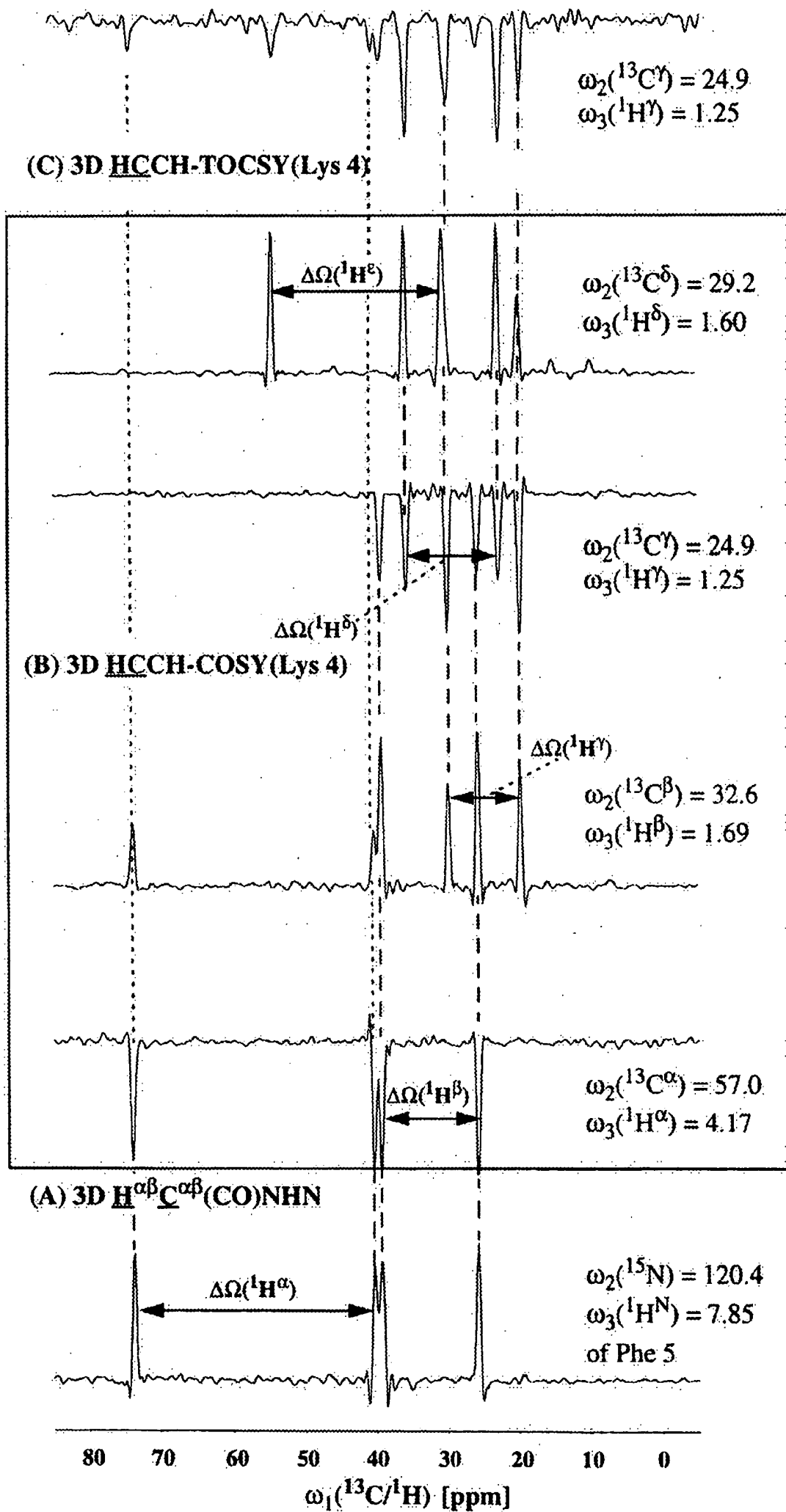
‡ The mixing time for the ^1H -TOCSY relay was set to 25 ms. The acquisition of central peaks is prevented by the use of spin-lock purge pulses (flanking the total correlation relay) to obtain pure phases.

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2D ^1H -TOCSY-relayed HCH-COSY

